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AN IMPROVEMENT TO A METHOD FOR MEASURING THE ABSORPTION COEFFICIENT OF ATMOSPHERIC DUST AND OTHER STRONGLY ABSORBING POWDERS

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A method, described in a previous publication, permits the measurement of the optical absorption coefficient of strongly absorbing particulate matter such as atmospheric dust. This is done by mixing the sample with a powder, and measuring the resulting degradation of the diffuse reflectance of the powder. This report shows how the spectral range, over which the method is applicable, may be extended by accounting for absorption of light in the weakly absorbing powder used a diluent. An example of the application of the method to an atmospheric dust sample is presented, showing that satisfactory measurements can be made

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from 0.3 to about 1.6 micrometers, using $BaSO_4$ as a diluent.

BaSO4

INTRODUCTION

The absorption coefficient of atmospheric dust is a quantity of considerable importance in current efforts to understand the behavior of light propagating in the earth's atmosphere. Methods for measuring this quantity are not straightforward, because of the difficulties involved in separating the light attenuated by a sample into scattered and absorbed components and then relating the absorbed energy to the optical absorption coefficient. Diffuse reflectance spectroscopy, and in particular the Kubelka-Munk (K-M) theory, can provide such information. A convenient method for using a laboratory spectroreflectometer to measure the K-M absorption coefficient of atmospheric dust has been described in an earlier publication [1]. The wavelength range over which the method is applicable is restricted by the optical properties of available powdered materials which are needed to dilute the sample. The purpose of this report is to show how this limitation can be alleviated by accounting for absorption of light in the diluent, thus extending the useful spectral range of the dilution method.

THEORY

In the procedure described earlier [1] a sample of dust is mixed with a finely powdered diluent, placed in a sample dish, and the diffuse reflectance is measured. The absorption coefficient is determined from the expression (Eq. 5 of Ref. 1)

$$k = s^* \frac{W}{W^*} F(R_\infty^*) \tag{1}$$

where

$$F(R_\infty^*) \equiv \frac{(1-R_\infty^*)^2}{2\, R_\infty^*} \tag{2}$$

is the K-M function. The details are described in the original report and the notation used here is consistent with that described in the earlier work.

Eqs. (1) and (2) are based on the assumption that the dust sample is the only absorber present, that is, the absorption coefficient of the diluent is zero. Since, in practice, no material is completely free of absorption, a diluent whose absorption coefficient is negligible compared to that of the diluent must be chosen. Thus, the wavelength interval over which the method is useful depends on the availability of materials with sufficiently small absorption coefficient to be used as diluents. A highly refined form of $BaSO_4$ has been found to be satisfactory for measurements on atmospheric dust samples in the 0.35 to 1.1 micrometer spectral interval. At longer or shorter wavelengths the intrinsic absorption in the $BaSO_4$ cannot be considered negligible.

In order to account for the effect of diluent absorption, one assumption about the physics of the optical situation must be made. We shall assume that when we are dealing with a mixture of powdered materials each component contributes linearly to the absorption coefficient of the mixture, in proportion to its molar concentration and individual absorption coefficient. This is analogous to the assumption (Bouguer-Lambert-Beers Law) that the effective absorption coefficient of a substance in liquid solution is directly proportional to its concentration in the solvent. In the case of solutions the linear relationship between concentration and absorption coefficient is strictly valid only for weak solutions; deviations from linearity can occur if the absorber concentration is too high.

For powdered samples, diluted with a non-absorbing (white) powder, similar effects might be expected. However, this is not a problem here. In this work the concentration of sample in the diluting powder is deliberately made small, on the order of one part in 10^2 to 10^4 by volume. In the dilution method for measuring the absorption coefficient of a strongly absorbing sample the requirement for weak concentration of sample is also dictated by the assumption that the scattering properties of the diluent are not significantly altered by the presence of the absorbing sample [1,2]. Suppose we consider a mixture of n absorbing powders, each with its own K-M absorption coefficient, k_n. The coefficient k_n represents the value that would be exhibited by the nth component if it were possible (which of course, it is not) to measure it in its pure form, undiluted by any other powder, or by voids between particles. If we assume that each component contributes to light absorption in the mixture in proportion to its concentration, then the coefficient k_n must be multiplied by the ratio of C_n*, the molar concentration in the mixture, to C_n, the molar concentration of the nth component in its pure, undiluted state. Then k*, the K-M absorption coefficient of the mixture of n component powders, can be described by an expression of the form

$$k^* = \sum_n (C_n^*/C_n)k_n = \sum_n (W_n^*/W_n)k_n, \qquad (3)$$

where W_n^*/W_n is the weight of nth component in the mixture divided by the weight of undiluted nth component required to fill the sample volume during the measurement, as described earlier [1].

In order to measure k for a sample of atmospheric dust we mix a small weight W* of sample with a much larger weight W_d^* of diluent. The two thoroughly mixed powders are then placed in a sample dish for diffuse reflectance measurement. The weights W and W_d of pure sample and pure diluent that would be sufficient to fill the sample dish can be calculated from the volume of the dish and appropriate specific gravities. The diffuse reflectance of the mixture, R_∞^*, is then measured, and its absorption coefficient k* is calculated from the K-M equation

$$k^* = s^* F(R_\infty^*) \tag{4}$$

where $F(R_\infty^*)$ is defined by Eq. (2). Using Eq. (3) above to describe k^* in terms of its two individual components, we can write Eq. (4) in the form

$$k^* = s^* F(R_\infty^*) = (W^*/W)k + (W_d^*/W_d)k_d \tag{5}$$

The last term in Eq. (5) can be determined from a separate measurement. One can put pure $BaSO_4$ powder in the sample dish and measure $R_{\infty d}$, the reflectance of the diluent. The scattering coefficient s' of the pure diluent is presumed known, measured by methods described elsewhere [1,3]. Then we can use the K-M equation

$$k_d' = s' F(R_{\infty d}) \tag{6}$$

to calculate k_d', the absorption coefficient for the diluent in the dish. The quantity k_d' in Eq. (6) is not the same as k_d in Eq. (5). The absorption coefficient for the solid material is k_d whereas k_d' represents that of the pure material in its powdered state — diluted with air spaces. Regarding the diluent powder as a "one component" mixture in the sense of Eq. (3), then we can write Eq. (6) in the form

$$k_d' = (W_d^*/W_d)k_d = s^* F(R_{\infty d}) \tag{7}$$

In the above expression we have made the usual assumption [2] that $s' = s^*$, that is the scattering coefficient of the diluent is not significantly changed by the presence of a small amount of sample mixed with it.

By combining Eqs. (5) and (7), and solving for k, we have

$$k = s^*(W/W^*) \left[F(R_\infty^*) - F(R_{\infty d})\right], \tag{8}$$

where

$$F(R_\infty^*) \equiv (1-R_\infty^*)^2/2R_\infty^*. \tag{9}$$

and

$$F(R_{\infty d}) \equiv (1-R_{\infty d})^2/2R_{\infty d} \tag{10}$$

In Eq. (8) k is the absorption coefficient of the atmospheric dust material itself, the value it should exhibit if it were not diluted by any other material or by the air spaces inherent in its powdered nature. The Eqs. (8), (9) and (10) provide a means of obtaining k from a weight measurement W*, a predetermined value of W calculated from knowledge of the sample specific gravity and the volume of the sample dish, and a

diffuse reflectance measurement, R_∞^*. The reflectance $R_{\infty d}$ of the diluent and scattering coefficient s* are determined in advance by other methods. For one form of high purity $BaSO_4$, these data have been published previously [3,4].

The dilution method is a convenient technique for determining the K-M absorption coefficients of various strongly absorbing powdered materials, including atmospheric dust. This coefficient can then be used to estimate the imaginary refractive index of the material in question. However, considerable care is required in using the technique, because the choice of diluent, and the properties of the sample itself limit the spectral region in which the method is applicable. Eq. (8) is useful in clarifying this and estimating the reliability of the method for a particular combination of sample and diluent.

The right member of Eq. (8) consists of two terms, one due to absorbance of light in the sample, and a second which expresses the effect of the presumably small absorption in the material chosen as a diluent. If the absorption coefficient k_d of the diluent is negligibly small, then the reflectance $R_{\infty d}$ will be nearly unity, and from Eq. (2) the quantity $F(R_{\infty d})$ is small. In the limiting case, as k_d approaches zero, Eq. (8) reduces to Eq. (1). Thus, the second term in Eq. (8) can be thought of as a correction term, and is only useful when the uncertainty in predetermined knowledge of $F(R_{\infty d})$ is negligible compared to the magnitude of $F(R_\infty^*)$.

The value of $F(R_{\infty d})$, at a given wavelength, is fixed once a diluent has been chosen, so the experimenter has no further control over it. The quantity $F(R_\infty^*)$ however, can be modified by measurement conditions, since R_∞^* depends on how concentrated the sample is. The sample concentration, or in practice the ratio W/W*, can be varied over a considerable range, but the assumptions made in the theory limit the choices somewhat. Examination of Eq. (8) shows that it is clearly desirable to cause $F(R_\infty^*)$ to be as large as possible, which means R_∞^* should be small. This is accomplished by making the sample as concentrated as possible. Earlier, however, we assumed that the absorption process was linearly dependent on concentration, and also that the scattering coefficient of the diluent was not appreciably changed by the presence of the sample. These assumptions limit the concentration of the sample, and therefore establish a minimum value for the choice of W/W*. In practice, for the $BaSO_4$ used in this work with atmospheric dust, W/W* should not be much less than 10^3. Because of this, the dilution method is useful only for strongly absorbing samples. If a sample has a low absorption coefficient, then unacceptably high concentrations are required to make the first term in Eq. (8) dominate the second term.

The above considerations suggest that the method should work best for extremely strong absorbers, because then $F(R_\infty^*)$ can be very large, even

without making W/W* unusually small. This is indeed the case, but one must keep in mind the limitations of diffuse reflectance measuring techniques. Most integrating sphere type measurements are less accurate when the measured reflectance is exceptionally low. So the characteristics of the instrumentation must be considered, and a choice of W/W* on the order of 10^4 or more may be appropriate for an extremely strongly absorbing sample - such as carbon soot - in order to avoid an unreasonably low value of the reflectance R_∞^*. This point is discussed further by Kortum [5].

Atmospheric Dust: An Illustrative Example

Figure (1) shows the result obtained by applying the methods described above to an atmospheric dust sample [6]. A quantity of 17.5 mg of dust was mixed with 15.8 gm of $BaSO_4$ diluent, and with the help of a clean glass plate about 4.5 gm of the mixture were pressed into a sample dish 3mm deep with a volume of 2.2 cm^3. Assuming the specific gravity of the dust to be 2.4, this results in a value of W/W* of 1095. The packing density should be about 2 gm/cm^3 for this choice of diluent. The reflectance R_∞^* was measured by usual methods. The same $BaSO_4$ diluent is also a good diffuse reflectance standard and was used as the reference material for all the reflectance measurements. The absolute reflectance of this standard has been published by Grum and Lucky [4], and these data were also used for the reflectance $R_{\infty d}$ of the pure diluent. The scattering number, s*, for the $BaSO_4$ was taken from the work of Gillespie, et al. [3]. The solid curve in Fig. (1) is the absorptive coefficient k, calculated from the first term only of Eq. (8), the result obtained without considering the intrinsic absorption of light in the $BaSO_4$. Note that the effect of diluent absorption in the visible spectrum is trivial, but that at wavelengths longer than about 1.2μm the effect of the $BaSO^4$ is important.

The error bars in Fig. (1) on the plot of k, calculated from Eq. (8) represent the result of an uncertainty in the input photometric data of 0.5% in the visible increasing to 1.0% in the infrared. It is clear from the figure that Eq. (8) can produce meaningful results even at wavelengths where the diluent absorbs some light, thus making possible measurements over a wider spectral interval than was possible with the previously published equation [1]. The exact spectral interval depends on the nature of the sample itself, as discussed above.

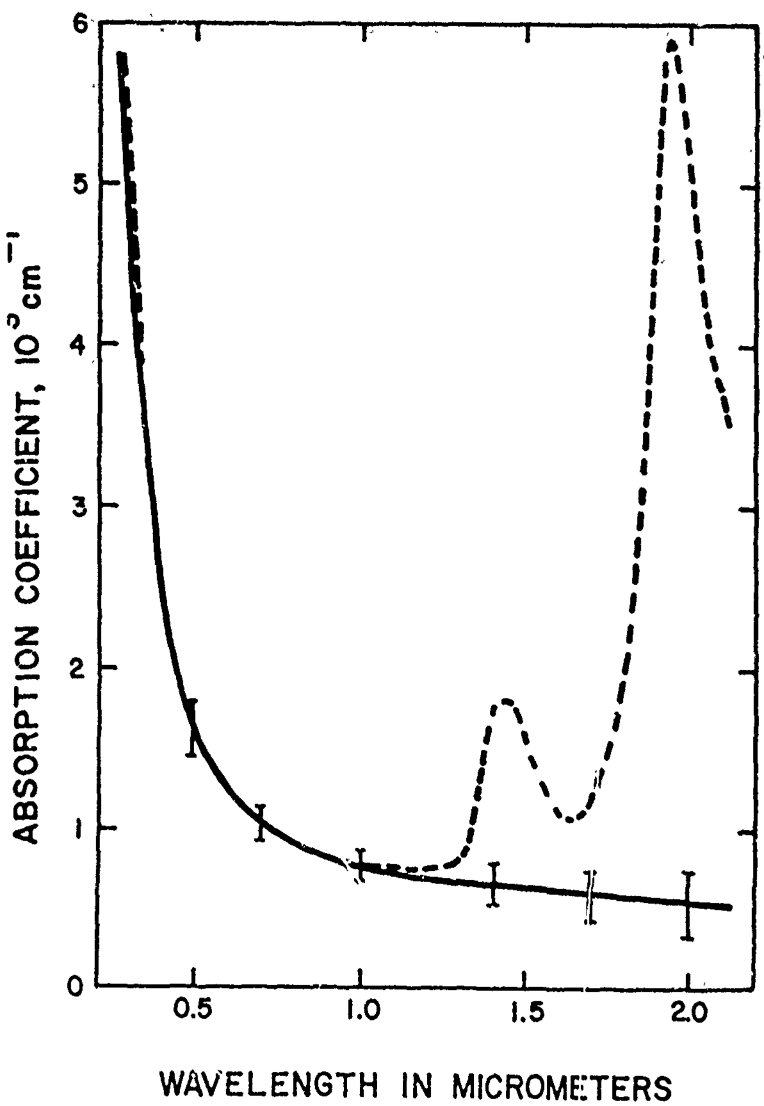

Figure 1. Absorption coefficient of atmospheric dust sample (solid curve) calculated from Eq. (8). The dotted curve represents the first term only of Eq. (8), the result obtained without accounting for the absorption of light in the diluting agent. The solid curve has been smoothed to eliminate spectrophotometric noise.

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